An Assessment of a PCB Analysis Procedure and Subsequent Estimation of Levels in Local Samples of Herring *(Clupea harengus harengus)*, Mackerel *(Scomber scombrus)*, Cod *(Gradus morhua)*, Halibut *(Hippoglossus hippoplossus)* and Eel *(Anguilla rostrata)*

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AN ASSESSMENT OF A PCB ANALYSIS PROCEDURE AND SUBSEQUENT ESTIMATION OF LEVELS IN LOCAL SAMPLES OF HERRING (*CLUPEA HARENGUS HARENGUS*), MACKEREL (*SCOMBER SCOMBRUS*), COD (*GADUS MORHUA*), HALIBUT (*HIPPOGLOSSUS HIPPOPLOSSUS*) AND EEL (*ANGUILLA ROSTRATA*).

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ABSTRACT

Hamilton, Erin and M. W. Gilgan. 1984. An Assessment of a Polychlorinatedbiphenyl (PCB) Analysis Procedure and Subsequent Estimation of Levels in Local Samples of Herring (Clupea, harengus harengus), Mackerel (Scombev scombrus), Cod (Gadus morhua), Halibut (Hippos glossus hippoglosous) and Eel (Anguilla rostrata). Canadian Technical Report of Fisheries and Aquatic Sciences No. 1215 : 9 pages.

A brief study was conducted to select a suitable PCB analytical procedure based on an existing well established extraction, fractionation and gas-chromatographic analytic procedure, one which was as simple as was compatible with reliability and which would be readily adaptable to pesticide studies. The method of Addison et al (1972) was adopted with minimal modification.

After a brief confirmation of the validity of the method it was used to determine the PCB content of tissues from selected fish species for comparison with results obtained eight to ten years previously from fish from the Maritime Provinces.

Due to severe time limitation the sampling was small. Muscle samples from mackerel, herring, cod, eel and halibut and liver samples from cod and halibut were analyzed. All sample PCB values except those from mackerel were lower than those reported by Sims et al (1977) or Zitko et al (1972) and (1974). In the mackerel the maximum PCB value found here (0.74 µg/g) was less than the maximum reported by Sims et al (1977) (0.93) for the same species. Overall the results suggest that PCB levels are declining in fish caught in or near Nova Scotia. Differences in analytical methods and sample size and diversity limit the validity and generalization of comparison.

Key words: Herring, Mackerel, Cod, Halibut, Eeel, PCB Analysis)

RÉSUMÉ

Hamilton, Erin et M.W. Gilgan, 1984. Évaluation d'une méthode de dosage des polychlorobiphényles (P.C.B.) et estimation de la concentration de ces produits dans des échantillons d'origine locale de hareng (Clupea harengus harengus), maquereau (Scombev scombrus), morue (Gadus morhua), flétan (Hippos glossus hippoglosous) et anguille (Anguilla rostrata). Rapport technique canadien des sciences halieutiques et aquatiques N° 1215 : 9 pages.

On a effectué une courte étude afin de trouver une méthode de dosage des P.C.B. satisfaisante qui repose sur des procédés d'extraction, de fractionnement et de chromatographie en phase gazeuse reconnus et qui soit simple et fiable en plus d'être facilement adaptable à l'étude des pesticides. La méthode d'Addison et coll. (1972) a été retenue et n'a nécessité qu'un minimum de modifications.

Après avoir rapidement vérifié la validité de la méthode, on l'a employée pour doser les P.C.B. présents dans les tissus de certaines espèces de poissons pour comparer ensuite les résultats obtenus avec des mesures effectuées 8 à 10 ans plus tôt sur des poissons provenant des Maritimes.

Comme le travail devait être fait dans un délai très court, l'échantillon était réduit. L'analyse a porté sur des échantillons de tissus musculaires de maquereau, hareng, morue, anguille et flétan et de tissus hépatiques de morue et de flétan. À l'exception des résultats obtenus pour le maquereau, les concentrations de P.C.B. mesurées étaient toutes inférieures aux valeurs données par Sims et coll. (1977) ou par Zitko et coll. (1972 et 1974). Dans le cas du maquereau, la concentration maximum de P.C.B. qu'on a mesurée (0,74 µg/g) était inférieure à la valeur maximum (0,93) donnée par Sims et coll. (1977). Dans l'ensemble, les résultats indiquent que la concentration des P.C.B. diminue chez les poissons pris en

Nouvelle-Écosse ou à proximité. Les différences de méthodes d'analyse et d'échantillons (effectif et variété d'espèces) limitent la validité de cette comparaison ainsi que la possibilité d'en généraliser les conclusions.

Mots-clés : hareng, maquereau, morue, flétan, anguille, P.C.B. (dosage)

INTRODUCTION

Polychlorinated biphenyls, or PCBs, were first produced in 1929 by the Swann Corporation for use as heat transfer media, especially in electrical transformers (1). These compounds are non-flammable and have excellent thermal stability; they are highly resistant to corrosive chemicals, acids and bases; they are insoluble in water and have a low vapour pressure at room temperature (2). Because of these properties, PCBs have been widely used as insulating fluids in electrical transformers and capacitors and in hydraulic fluids and heat transfer systems. They have also found application in the preparation of consumer goods such as plastics, adhesives, paints and varnishes, printing inks, carbonless copy paper and in solutions to dissolve pesticides (3).

PCBs may enter the environment in a number of ways. They may be released from damaged or rusted electrical equipment or through spillage and then may seep into streams, rivers and lakes. They may also be poured directly into sewage drains. If improperly incinerated, PCBs can become airborne, dispersed by the wind and deposited to earth in rain or snow.

The first identification of PCBs as an environmental contaminant was made by Jensen in 1966 (4), who found the compounds in extracts of fish and bird samples. They have since been identified in fish, birds, animals and sediments all over the world, and also have been found in human adipose tissue and breast milk (5).

In 1968 an accident occurred in Japan which focused attention on the potential problem of PCB contamination of food. Rice oil became contaminated when PCBs leaked from a heat exchanger. People consuming the contaminated oil exhibited several symptoms such as chloracne, discoloration of the gums and nailbeds, swelling of joints, lethargy and joint pains (6). This incident confirmed that the health of humans could be adversely affected by PCBs.

The high stability and lipid solubility of PCBs result in the accumulation of these substances in the food chain. They may persist and become concentrated in foods eaten by man to levels which are many times greater than environmental background levels. Thus, the concentration of PCBs in foods must be monitored to ensure that the foods are safe for consumption.

PCBs have been known to be present in fish and seals of Atlantic Canada since 1967. Holden and Marsden (7) studied the occurrence of chlorinated pesticides (DDT and related compounds) in blubber of grey seals and harp seals on the Canadian Atlantic coast. They found indications that certain unidentified compounds which were present in the sample extracts were PCBs, based on a comparison of the gas chromatographic patterns produced by the unknowns and commercial PCB formulations.

Addison, Zinck and Ackman (8) reported levels of organochlorine pesticides and PCBs in Canadian marine oils. Herring oils produced in the late 1960's from the Gulf of St. Lawrence herring contained up to 11 ppm PCBs, with oils from fish caught in the spring giving higher values than oils from fish caught in autumn.

Concentrations of PCBs in fish, cormorants and herring gulls from the Bay of Fundy - Gulf of Maine area were reported by Zitko et al (9). Some examples of fish and the levels found (in µg/g) include the following: herring (0.34), mackerel (0.35), ocean perch (0.32), cod (0.55), bluefin tuna (1.54) and herring oil (3.55).

Zitko et al (10) described levels and distribution of PCBs in Atlantic herring originating in the Gulf of St. Lawrence and Bay of Fundy, and also in yellow perch from New Brunswick rivers. Mean PCB concentrations for Gulf of St. Lawrence fish were 0.31 µg/g and for Bay of Fundy fish were 0.64 µg/g. Values for yellow perch ranged from 0.089 to 0.13 g/g.

Results of a survey of PCBs in fish and fisheries products from the Atlantic provinces were reported in 1977 by Sims et al (11). This fish had been collected in 1971 and 1972 and included 29 species of crustacea, bivalves and finfish, as well as 7 selected tissues and fishery products. It was found that PCBs were accumulated preferentially in lipid-rich specimens. Some mean concentrations found (values in µg/g) include: lobster (0.098), cod (0.040), halibut (0.27), eels (0.44), herring (0.52), mackerel (0.41) and cod liver (5.1).

The purpose of this study has been to develop the capability to analyze fish and fisheries products for PCBs, using gas chromatography as a measurement technique. It was also intended to apply the method in the analysis of actual samples for inspection purposes. Details of the method development and results obtained from a small survey of selected samples will be presented in sections of this report.

MATERIALS AND METHODS

Hexane ("Distilled in Glass" grade from Caledon) was re-distilled in an all-glass apparatus. The chloroform and methanol were ACS reagent grade from Fisher Scientific; the methanol was distilled before use and the chloroform was used as received. Florisil, 60-100 mesh was PR (Pesticide Residue) grade from the Floridin Company. This was removed from the original container and stored in pre-cleaned glass jars with foil-lined screw tops. The Florisil was activated in beakers in 20 g portions in an oven at 135°C until used (at least 16 hours). Anhydrous sodium sulfate, 12-60 mesh, from J.T. Baker Company was hexane-washed, air-dried and stored in a glass jar with a foil-lined top.

Standard solutions of PCBs were prepared using Aroclor 1254 and Aroclor 1260 from Analabs Ltd.

Gas chromatography was carried out on a Perkin-Elmer Model 3920B gas chromatograph equipped with a 63 Ni Electron Capture Detector. Two different packed columns were used. The first was a 6 ft. long, 2 mm I.D. column packed with 1.5% OV-17 plus 1.95% QF-1 on Chromosorb W (HP) support, 80-100 mesh. Later a second column was used to confirm the identity of the sample peaks. This was also 6 ft, 2 mm I.D., packed with 4% SE-30 plus 6% QF-1 on Chromosorb W (AW), 80-100 mesh. Argon/Methane (95%/5%) was the carrier gas. The operating temperatures for Injector, Column, Interface and Detector were 200°, 190°, 240° and 240°C respectively.

An adaptation of the method of Addison et al (8) for the determination of PCBs in fish samples was developed and applied as follows: 10.00 g of homogenized fish samples were blended with 150 milliliters of 2:1 chloroform/methanol using a Brinkmann Polytron. The mixtures were then filtered into separatory funnels and distilled water was added to give final proportions of 8:4:3 for volumes of chloroform, methanol and water, respectively, including the amount of water contributed by the sample. The mixtures were shaken vigorously for one minute and allowed to stand overnight to clear.

The chloroform phase was drawn off into a flask and about 30 g of anhydrous sodium sulfate was added to dry the solution. The liquid was then filtered into a round-bottomed flask and evaporated using a rotary evaporator. After removal of chloroform, an oily residue remained. Several additions of a few milliliters of hexane, followed by evaporation of the solvent, were made to ensure removal of all chloroform and to change the solvent to hexane. The residue was then quantitatively transferred to a 10.0 mL volumetric flask and diluted to the mark with hexane.

Florisil columns were prepared by pouring 20 g of activated absorbant into glass chromatographic columns (300 mm long and 20 mm I.D.) containing hexane. The columns were topped with about 1 cm of anhydrous sodium sulfate. Aliquots of sample extract containing 300-400 mg of lipid were pipetted onto the top of the columns. The columns were then eluted with 200 mL of hexane at a flow rate of approximately 1 mL/min. The column eluates were concentrated to a few milliliters, then diluted to 5.0 mL with hexane to give solutions for GC analysis.

Samples of several different kinds of fish were obtained and analyzed for the presence of PCBs to apply the procedure and also as an informal "mini-survey" of PCB levels in these fish.

Six mackerel were obtained which had been caught off Eastern Passage, N.S. in late November, 1981. Six herring were supplied from a lot caught in Herring Cove in the months of August and September, 1981. The herring and mackerel were filleted and the fillets were homogenized and stored frozen until ready for analysis.

Five large cod were supplied from Eastern Passage in January, 1982. These were weighed, filleted and their livers were removed. The fillets and livers were homogenized and stored frozen. Six eel samples were received which had been taken from Pictou Harbour and inlet streams in early February, 1982. These were skinned and the fillets were homogenized. Finally, five fresh halibut were received from Sambro in mid-March. As was done for the codfish, samples of the fillets and livers were homogenized and stored for analysis.

All homogenized samples were stored in a freezer in glass jars with foil-lined tops. When ready for analysis they were thawed and re-mixed to ensure homogeneity. The sample size used for analysis was 10.00 g for fillets and 2.00 g for liver samples. All samples were analyzed in duplicate. Based on the appearance of the peaks in the gas chromatograms of the sample extracts, the PCBs were quantified as either Aroclor 1254 or Aroclor 1260.

ASSESSMENT AND OPTIMIZATION OF PROCEDURE

Homogenized herring tissue samples were used to assess the procedure and optimize the recovery of added spikes of PCBs at various stages.

After an initial trial of the procedure was made, an extract of 10.00 g tissue was prepared. The chloroform extract was diluted to 10.0 mL in a volumetric flask. 3.0 mL of lipid extract were mixed with 1.0 mL of 10 µg/mL Aroclor 1254 and diluted to 10.0 mL with hexane. Two Florisil columns were prepared and 5.00 mL of the spiked lipid mixture was applied to each column. One column was eluted with 200 mL hexane, and one with 150 mL of 6% diethyl ether/hexane. The eluates were concentrated to 10.0 mL and injected into the GC. Five peaks were chosen for quantitation, their heights were measured and summed and these were compared with Aroclor 1254 standards.

Solution	Peak Height Sum (arbitrary units)
Column eluted with 200 mL hexane	87.5
Column eluted with 150 mL ether/hexane	85.0
Aroclor 1254, 1.0 µg/mL	175.5
Aroclor 1254, 0.5 µg/mL	87.8

The expected concentration of Aroclor 1254 in the final solution from each column was 0.5 µg/mL. As can be seen from the results, the recovery was quantitative, with little difference between elution with pure hexane and with the ether/hexane solution. It was decided to use hexane alone for the elution, due to the hazards of working with diethy ether.

To estimate the recovery of an added spike from the entire procedure, an extract of 10 g herring tissue was prepared with a spike of 50 µg. Aroclor 1254 added during the homogenization procedure. In the Florisil cleanup step, 1.5 mL of the lipid solution were applied to the column, which was then eluted with 200 mL of hexane. The eluate was concentrated to 10.0 mL and injected into the GC, along with standards. The sum of five peaks in standards and samples were compared to obtain the recovery.

Solution	Peak Height Sum (average of 2 injections)
0.5 µg/mL Aroclor 1254	104
1.0 µg/mL Aroclor 1254	191
2.0 µg/mL Aroclor 1254	397
Spiked extract	125

From the standard curve, the concentration of Aroclor 1254 in the eluate was found to be 0.65 µg/mL. Since the expected concentration would be 0.75 µg/mL, the recovery was found to be 87%.

Next, it was decided to prepare a mixture of organochlorine pesticides which might occur in fish samples to see if these could be separated from PCBs using a Florisil column, and also to see which peaks in the PCB chromatogram would not be useful for quantitation purposes due to possible overlap with interfering pesticides.

A solution of pesticides in hexane was prepared which contained the following compounds (concentrations indicated in parentheses are in units of µg/mL): lindane (0.01), heptachlor (0.02), aldrin (0.02), heptachlor epoxide (0.03), p,p'-DDE (0.02), o,p'-DDT (0.06), p,p'-DDD (0.06) and p,p'-DDT (0.03). See Figure 1 for a chromatogram of this mixture obtained under the previously stated operating conditions for the gas chromatograph.

An aliquot of the pesticide mixture was pipetted onto a Florisil column, which was then eluted with 200 mL hexane. From comparison of the chromatograms of the original standard and the concentrated eluate, it was found that 100% of the added aldrin, p,p'-DDE and o,p'-DDT, as well as 10% of the heptachlor, were eluted under these conditions, i.e. in the same fraction which would contain PCBs. Therefore, the PCB peaks which might overlap with these peaks in the chromatogram could not be used for quantitation purposes.

A PCB-spiked lipid extract, which had been spiked with Aroclor 1254 to a concentration of 4.3 µg/mL, was mixed with the pesticide mixture to see if the PCBs and pesticides could be separated and recovered from the Florisil column cleanup. Since 2.0 mL of the lipid extract were applied to the Florisil column and the eluate was concentrated to 10.0 mL, the expected concentration of Aroclor 1254 in the solution was 0.86 µg/mL. Injection of the solution and comparison of the sum of four peak heights to standards indicated a concentration of 0.82 µg/mL, which corresponded to a recovery of 95%.

RESULTS AND DISCUSSION

It was possible to separate PCBs from several organochlorine pesticides as part of a Florisil column cleanup step. This was achieved both for standard mixtures and for spiked extracts of fish samples. Recoveries of PCBs from the Florisil cleanup procedure were high, usually 95 to 100%. Recoveries from the entire procedure ranged from 80 to 92%. Corrections for estimated recovery are not often carried out in PCB analysis, since analysts are only able to estimate the PCB content of a sample and since there is no standard way of quantitating the residues. This is because the PCB chromatograph obtained from a sample extract is never exactly the same as the trace obtained from a standard, so that one must estimate which PCB mixture is present. The sum of the heights of four prominent and/or interference-free peaks has been used in this work to quantitate the PCBs. Again, this is a matter of choice, as others have used many different means of quantitation such as the heights of five peaks of Aroclor 1254 (10), or the heights of "all peaks occuring after peak #7 (inclusive) of Aroclor 1254, using the numbering system of Reynolds" (11).

SURVEY OF PCBs IN FISH SAMPLES

Aliquots of the cleaned-up sample extracts from the procedure were injected into the gas chromatograph using the operating conditions stated earlier. Replicate injections were made until satisfactory precision was achieved. A series of standards for each PCB standard mixture (Aroclor 1254 and Aroclor 1260) was prepared; the final concentrations of these were 0.10, 0.20, 0.50 and 1.0 µg/mL. The Aroclor standard whose gas chromatograph matched more closely to the sample chromatogram was used for quantitation. Four peaks were selected for use in quantitation on the basis of freedom from possible interferences due to organochlorine pesticides. Figure 2 shows a chromatogram of Aroclor 1254, Figure 3 a trace of Aroclor 1260 and Figure 4 a halibut liver extract with the peaks used for quantitation indicated. All these chromatograms were obtained using the OV-17/QF-1 column.

Quantitation of PCBs was done by measuring the heights of these four peaks and taking the sum of the peak heights, for both the samples and standards. Plots of peak height sum against PCB concentration were prepared for the standards. The linear regression technique was used to fit the best straight line to the standard plot. Values for the peak height sums for the samples were then inserted into the equation for the line and the PCB concentrations were then corrected for dilution factors to give the value for the original sample in units of µg/g (or ppm). The mean of determinations for two replicate analyses of each fish sample was reported. All samples were quantified as Aroclor 1254, with the exception of the eel samples, which were quantified as Aroclor 1260.

Tables I to V give the concentrations found for the samples analyzed. Concentrations are expressed in µg/g and are the average of two results for each sample.

TABLE I

PCBs In Mackerel Muscle
(all similar in size, about 16 in. long)

Sample No.	PCB concentration, µg/g
1	0.42
2	0.66
3	0.29
4	0.56
5	0.70
6	0.74
MEAN ± SD	0.56 ± 0.18

TABLE II

PCBs In Herring Muscle
(all similar in size, about 12 in. long)

Sample No.	PCB concentration, µg/g
1	0.26
2	0.26
3	0.41
4	0.26
5	0.16
6	0.086
MEAN ± SD	0.24 ± 0.11

TABLE III

PCBs In Cod Muscle (1-5) Or Liver (6-10)

Sample No.	Description	PCB Concentration, µg/g
1	36 in long, 17 lb	N.D.
2	26 in long, 6½ lb	N.D.
3	27 in long, 7 lb	0.033
4	24 in long, 5 lb	0.020
5	25 in long, 4½ lb	0.038
MEAN ± SD		0.02 ± 0.02
6	Liver of No. 1	3.63
7	Liver of No. 2	2.32
8	Liver of No. 3	2.55
9	Liver of No. 4	2.78
10	Liver of No. 5	2.86
MEAN ± SD		2.83 ± 0.50

N.D. - not detected

TABLE IV

PCBs In Eel Muscle

Sample No.	Description	PCB Concentration, µg/g
1	26½ in long, 1.1 lb	0.28
2	25 in long, 0.87 lb	0.56
3	24¼ in long, 1.0 lb	0.36
4	22 in long, 0.69 lb	0.087
5	23 in long, 0167 lb	0.27
6	23 3/4 in long, 0.66 lb	0.18
MEAN ± SD		0.29 ± 0.16

TABLE V

PCBs In Halibut Muscle (1-5) Or Liver (6-10)

Sample No.	Description	PCB Concentration, µg/g
1	39 in long, 42.5 lb	N.D.
2	35 in long, 19.4 lb	0.023
3	34 in long, 35.5 lb	0.037
4	38 in long, 25.3 lb	0.077
5	37 in long, 18.7 lb	0.018
MEAN ± SD		0.031 ± 0.029
6	Liver of No. 1	0.54
7	Liver of No. 2	0.96
8	Liver of No. 3	0.68
9	Liver of No. 4	0.54
10	Liver of No. 5	1.38
MEAN ± SD		0.82 ± 0.36

The highest PCB levels were found for cod livers (Table III), ranging from 2.32 to 3.63 µg/g. These results indicate that fish having fatty tissue such as mackerel, herring and eel, have higher PCB concentrations than halibut or cod, which have leaner flesh.

To confirm the identity and concentrations of PCBs in samples, it is desirable to use a second packed column in the gas chromatograph which has different retention characteristics for PCBs. In this work, a column packed with 4% SE-30 plus 6% QF-1 on chromosorb W (HP) support was installed. Standards and selected sample extracts were injected and the PCB content of the samples was calculated as described earlier. The results are shown in Table VI.

TABLE VI

Comparison of PCB Estimates After Separation on Two Common GC Column Packings

Sample	OV-17/QF-1 Column	SE-30/QF-1 Column
Mackerel No. 1	0.42	0.42
Mackerel No. 5	0.70	0.72
Herring No. 3	0.41	0.40
Herring No. 5	0.19	0.23
Cod (Flesh) No. 4	0.020	0.042
Cod No. 6 (liver)	3.63	3.84
Cod No. 8 (liver)	2.55	2.72
Halibut (flesh) No. 3	0.037	0.052
Halibut No. 9 (liver)	0.54	0.78

The agreement, in general, between the two sets of results is good. At very low levels differences are seen, but this is to be expected since at these low concentrations errors may be made in measuring the small peaks and these errors would be magnified by the correction factors for dilution.

Comparison of the results found in this small survey with those from fish caught in 1971-72 from the Northwest Atlantic (11) show present concentrations are generally lower (Table VII).

TABLE VII

Comparison Of The Levels Of PCBs From The Present Determinations And Those From A Previous Survey In This Area

		This Work		Sims et. al. (11)
Groundfish	N		N	
Cod	5	N.D.-0.038	20	0.012-0.091
Halibut	5	N.D.-0.077	10	0.065-0.60
Pelagic Fish				
Herring	6	0.086-0.41	9	0.27 -0.74
Mackerel	6	0.29 -0.74	23	0.093-0.93
Other				
Cod Livers	5	2.32 -3.63	11	0.89 -19.3
Eels	6	0.087-0.56	11	0.13 -1.2

N.D. - not detected

Comparisons between two groups of data must be made with caution, as the sample numbers are different and the sampled area in the previous work was more extensive. Also, different extraction and quantitation methods were used. However, it does appear that the levels of PCBs in the samples analyzed in this work are generally lower than in samples collected ten years ago. This may be due to declining use of PCBs as a result of government regulations.

CONCLUSION

A method for the determination of PCBs in fish and fishery products was adopted and applied. Recoveries of added spikes of PCBs for the entire procedure have generally been good, ranging from 80% to 92%. Corrections to the observed sample PCB concentrations have been made to take into account the estimated recovery. Although the entire method is time-consuming, multiple samples may be processed at the same time.

Further work could be done to extend the method to allow the determination of the organochlorine pesticides. This would require further elution of the Florisil column with a more polar solvent mixture, as hexane will not elute the polar pesticides. A more efficient gas chromatography column such as a glass or fused silica capillary column could be used to more effectively separate the peaks due to PCBs from those due to pesticides. This would reduce possible interferences from overlapping peaks and would permit quantitation of low pesticide levels.

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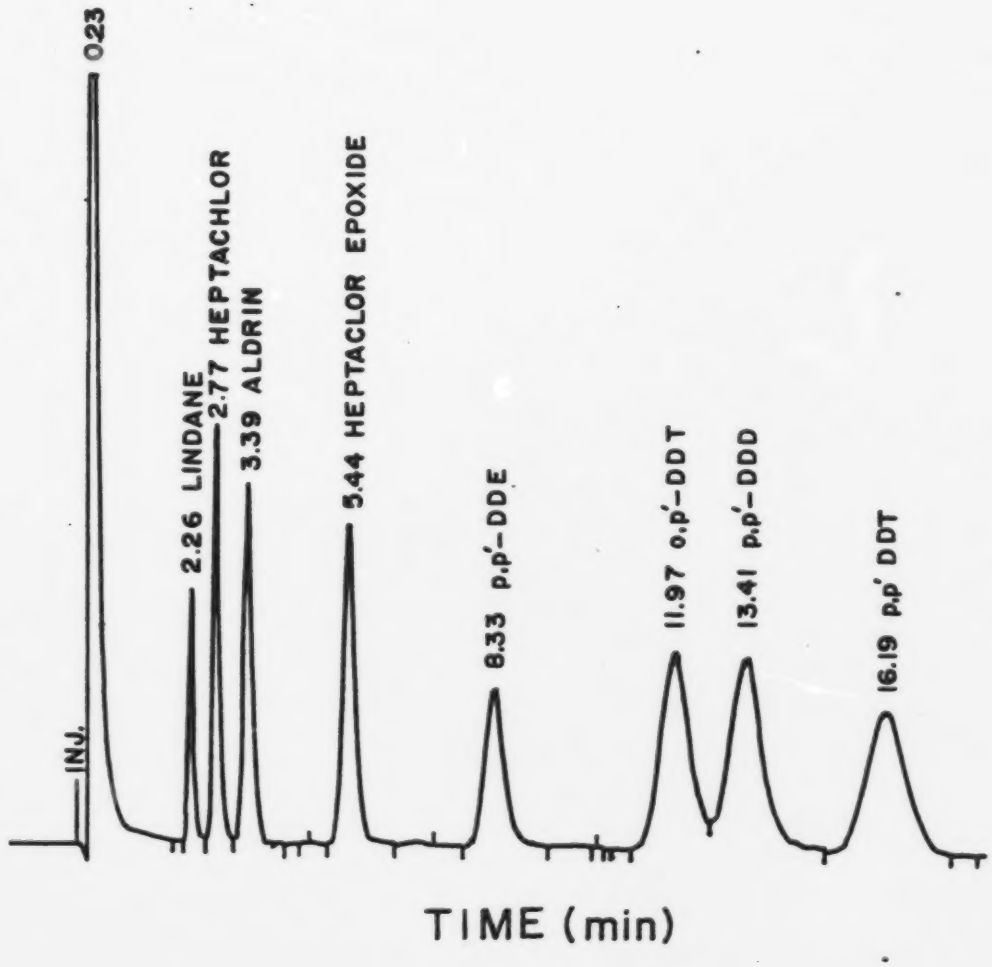

FIGURE 1 GC chromatogram of the standard pesticide mixture on an OV-17/QF-1 column

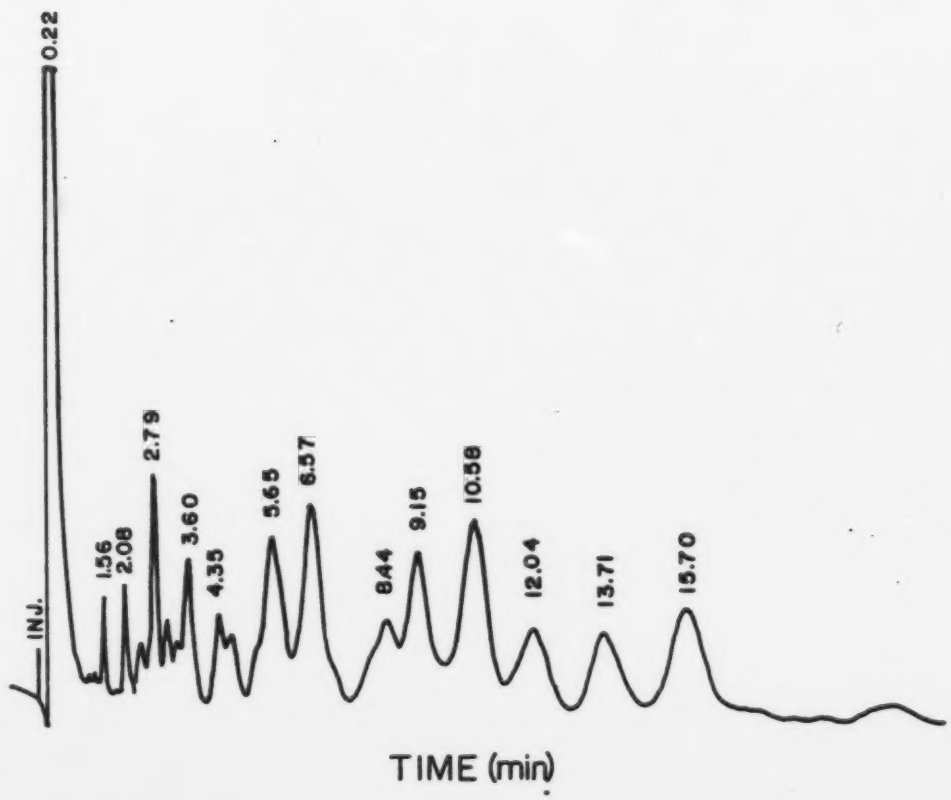

FIGURE 2 GC chromatogram of the standard Aroclor 1254 mixture on an OV-17/QF-1 column

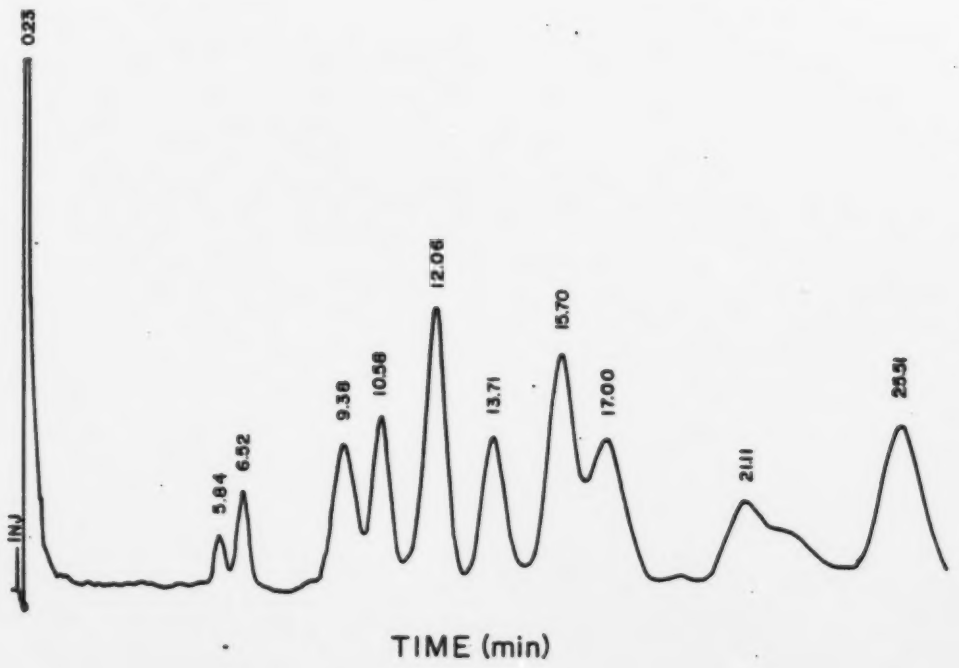

FIGURE 3 GC chromatogram of the standard Aroclor 1260 mixture on an OV-17/QF-1 column

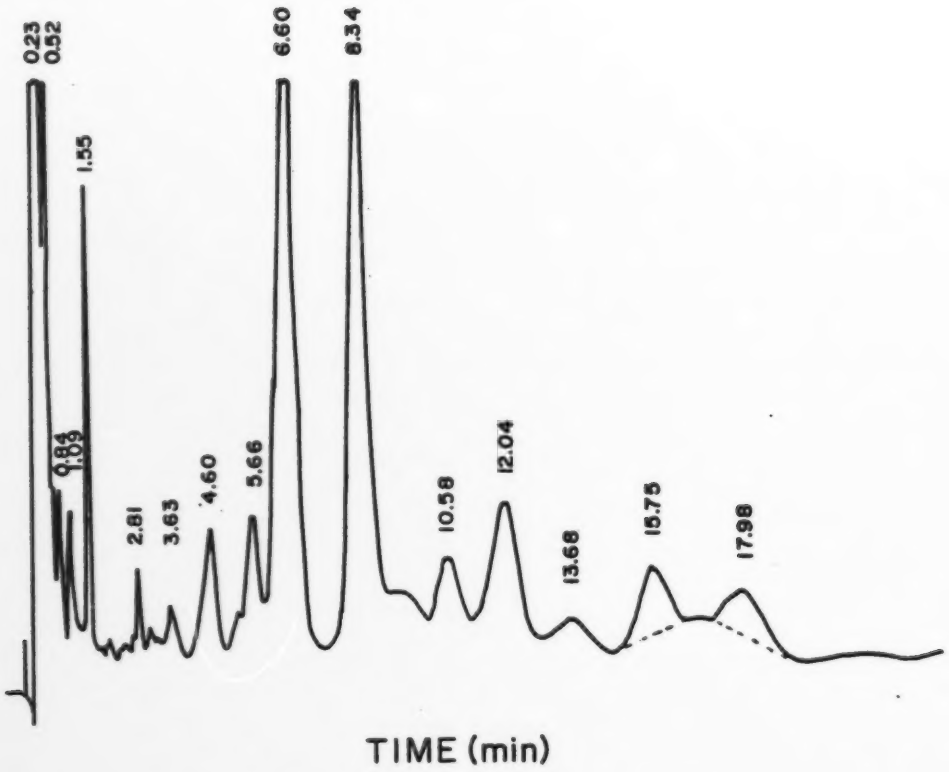

FIGURE 4 GC chromatogram of a halibut liver extract on an OV-17/QF-1 column. Peaks at 5.66, 10.58, 13.68 and 15.75 minutes were used for quantitation.